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09/901,975 CH2782 US NA

Response to Office Action dtd. 8/20/03

Rule 132 Declarations by: Jon L. Howell (Inventor)

Darryl DesMarteau (Professor of Clemson University)

Joseph Thrasher (Inventor/Professor of University of Alabama)

Patents:

Note:

US 4,523,039 GB 1,104,482

EPO 0 148 482 B1 EPO 0 340 793 B1 EPO 0 344 547 A2

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### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:

Jon L Howell et al

CASE NO.: CH2782 US NA

SERIAL NO.: 09/901,975

**GROUP ART UNIT: 1714** 

FILED: 07/10/2001

**EXAMINER:** Cephia Toomer

FOR: Thermally Stable Perfluoropolyethers and

Processes Therefor and Therewith

#### **RULE 132 DECLARATION**

Commissioner for Patents P. O. Box 1450 Alexandria, VA 22313-1450

Sir:

I, Jon L. Howell, declare that:

I am a citizen of the United States of America residing in Bear, Delaware;

I am a co-inventor of the above-identified application for a US patent and have read the references cited by the examiner.

I received my B. S. degree in Chemistry from The University of Missouri at Rolla in 1972, and a Ph.D. degree in Inorganic Chemistry of the Non-metals, including emphasis in fluorine chemistry from Virginia Polytechnic Institute and State University in 1978; and

I was employed by Duke University as a Postdoctorial Associate from 1978 to 1980; and have been working for E. I. du Pont de Nemours and Company ("DuPont") since 1980, during which time, I have been working in the area of Organic and Inorganic Fluorine Chemistry, and sole supporting organofluorine chemist for the Performance Lubricants Business within DuPont and specializing fluoropolyether chemistry.

therefore, I am very knowledgeable in all perfluoropolyether products ever produced by DuPont; and

to the best of my knowledge, DuPont has never been able to produce any perfluoropolyethers comprising repeat units of O-(CF(CF<sub>3</sub>)CF<sub>2</sub>-O)<sub>w</sub> that contain Rf and Rf' having 3 carbon atom segments essentially free of C<sub>2</sub> units as disclosed in Moggi (column 3,

lines 32-47) until I and my co-inventors discovered the perfluoropolyethers as disclosed and claimed in the above-identified patent application.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Jon L. Howell

11/20/03

Date

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PATENT

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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FOR: Thermally Stable Perfluoropolyethers and

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#### **DECLARATION**

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

I, Darryl D. DesMarteau, declare that:

I am a citizen of the United States of America residing in Clemson, South Carolina; I received my Ph.D. degree in Chemistry from The University of Washington in 1966;

I was an Alfred P. Sloan Fellow (1975-77) and an Alexander von Humboldt Research Fellow (Heidelberg, 1979-80). I received the ACS Award for Creative Work in Fluorine Chemistry, sponsored by PCR, Inc. (1983), the Clemson University Alumni Award for Outstanding Research (1985), Governor's Award for Excellence in Science Research (1988), the Alexander von Humboldt Senior Distinguished U.S. Scientist Award (1989), the ACS Charles H. Stone Award (1993), Washington State University Alumni Achievement Award (1995) and the College of Engineering and Science Award of Excellence for Faculty Achievement in the Sciences (1996). I serve on the Board of Editors of the Journal of Fluorine Chemistry.

I have been teaching chemistry at Clemson University, Clemson, South Carolina since 1982 and hold the Tobey-Beaudrot Professor of Chemistry endowed chair;

as professor, I have also been extensively conducting research in fluorocarbon chemistry, especially in perfluoropolyethers produced by the worlds main perfluoropolyether producers, namely E.I. du Pont de Nemours and Company, USA, Daikin Industries, Japan, and Ausimont, S.p.A, Italy;

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based on my 37-year experience in fluorocarbon chemistry including perfuoropolyethers, I further declare the following;

I have read the references, Caporiccio (US 5,217,123) and Moggi (US 5,077,097), cited by the USPTO;

with respect to those regarding R'f end groups in structures c and f disclosed in Caporiccio, which are disclosed as available from DuPont of USA and Daikin Industries of Japan, respectively, it is my opinion that it is impossible for R'f to be independently selected from -CF<sub>3</sub>, -CF<sub>2</sub>CF<sub>3</sub>, or -C<sub>3</sub>F<sub>7</sub> based on the information disclosed in Caporiccio for producing these perfluoropolyethers;

for structures d, e, and g as available from Ausimont, S.p.A., the end groups would be  $-CF_3$  and  $CF_2CF_3$ , based on previous publications by Ausimont and, as far as I am aware a  $C_3F_7$  end group is not available in high molecular polymers from Ausimont meeting the required viscosity range disclosed in Caporiccio; and

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Darryl D. DesMarteau

DATE

College of Arts & Sciences
Department of Chemistry



PATENT

# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:

ALABAMA

ARTS & SCIENCES

Jon L. Howell, et al.

CASE NO.: CH2782 US NA

SERIAL NO.: 09/901,975

**GROUP ART UNIT: 1714** 

FILED: 07/10/2001

EXAMINER: Cephia Toomer

FOR: Thermally Stable Perfluoropolyethers and

Processes Therefor and Therewith

## RULE 132 DECLARATION

Commissioner for Patents P. O. Box 1450 Alexandria, VA 22313-1450

Sir:

I, Joseph S. Thrasher, declare that:

I am a citizen of the United States of America residing in Tuscaloosa, Alabama;

I am a co-inventor of the above-identified application for US patent;
I received my B.S. degree in Chemistry from Virginia Polytechnic
Institute and State University (Virginia Tech) in 1978, and a Ph.D. degree in
Inorganic Chemistry, with an emphasis on main-group and fluorine chemistry,
from Virginia Tech in 1981;

I was employed as a Postdoctoral Research Associate at the Freie Universität Berlin in Berlin, Germany in 1981 to 1983 and as a Visiting Assistant Professor at Clemson University in 1983 to 1984. In both locations, I again carried our research in the area of fluorine chemistry. In 1984 I joined the faculty at The University of Alabama, where I have risen through the academic ranks to my current position as Professor and Chair of Chemistry. During 1992 I spent an

eight-month sabbatical at the Experimental Station of E. I. du Pont de Nemours and Co., Inc.;

in terms of professional societies and honors, I am a member of the American Chemical Society, including the Divisions of Inorganic, Polymer, and Fluorine Chemistry. I have held a variety of elected, service positions in the latter including Past Chair (1995), Chair (1994), Chair-Elect (1993), and Executive Committee (1989-91). I am also a member of the Fluorine Chemistry International Steering Committee (1995-present), which decides on the locations for both European and International Symposia on Fluorine Chemistry. I have also been a consultant for both E. I. du Pont de Nemours and Co., Inc. (1993-1999, 2001-present) and Conoco, Inc. (1988-1990). I am a member of the Virginia Tech Department of Chemistry Advisory Council (2001-present); I am an Expert Analyst for Chemistry Advisory Council (2001-present); and I have served as Guest Editor of two memorial issues of the Journal of Fluorine Chemistry [1998, 89(1) and 1990, 48(3)]. I was an invited participant at the 2nd NSF Workshop on Main-Group Chemistry in 1984, and I also won a certificate of teaching excellence from Virginia Tech in 1980;

I have also organized or will organize the following symposia or conferences in the area of fluorine chemistry: Organizing Chairman of the 19th International Symposium on Fluorine Chemistry to be held in San Diego, CA in 2009; Co-Chairman of the ACS 12th and 11th Winter Fluorine Conferences, January 1995 and January 1993; Co-Organizer of a symposium entitled "Alternatives to Chlorofluorocarbons" for 46th Southeast Regional Meeting of the American Chemical Society, October 1994; Organizer of a symposium entitled "Fluorine and Fluorine-Containing Substituent Groups in Inorganic Chemistry" for the 203rd National Meeting of the American Chemical Society, April 1992. From the latter symposium I co-edited an American Chemical Society Symposium Series book entitled "Inorganic Fluorine Chemistry: Toward the 21st Century;"

over my 19+ year independent academic career I have generated over \$5 million in external support for my group's research in fluorine chemistry from

sponsors such as E. I. du Pont de Nemours and Co., Inc. (DuPont); National Aeronautical and Space Administration (NASA); Contract Research and Licensing, Inc. (CR&L); U.S. Department of the Navy, Naval Surface Warfare Center (NSWC); Minnesota Mining and Manufacturing, Inc. (3M); Monsanto Co.; American Chemical Society-Petroleum Research Fund (ACS-PRF); American Society of Heating, Refrigeration, and Air-Conditioning Engineers (ASHRAE); Air Products and Chemicals, Inc.; DowElanco, Inc.; U.S. Department of Energy (DOE); U.S. Department of Transportation (DOT); and U.S. Environmental Protection Agency (EPA) via Gulf Coast Hazardous Substances Research Center (GCHSRC). Much of this research has been in the area of fluorocarbon chemistry, especially in area of perfluoropolyethers as produced by the world's main perfluoropolyether manufacturers, namely E. I. du Pont de Nemours and Company, USA; Daikin Industries, Japan; and Ausimont, S.p.A, Italy;

based on my 22+ years of experience in fluorocarbon chemistry including perfluoropolyethers, I further declare the following:

I have read the references, Caporiccio (US 5,210,123) and Moggi, et al. (US 5,077,097), cited by the examiner;

and with respect to structures of the type shown in formulas (c - g) by Caporiccio, it is my educated opinion that  $R'_F$  cannot be independently selected (selectively chosen) from a list of perfluoroalkyl radicals having one to three carbon atoms, namely  $-CF_3$ ,  $-C_2F_5$ , and  $-C_3F_7$ . To be able to do so based on the art disclosed as available by Caporiccio would imply that one could choose a neat, clean perfluoropolyether from one of nine different possible combinations of end groups  $R'_F$  for each of the structures of the type shown by formulas (c - g), namely

$$R'_{F} = CF_{3}$$
 and  $R'_{F} = -CF_{3}$   
 $R'_{F} = CF_{3}$  and  $R'_{F} = -C_{2}F_{5}$   
 $R'_{F} = CF_{3}$  and  $R'_{F} = -C_{3}F_{7}$   
 $R'_{F} = C_{2}F_{5}$  and  $R'_{F} = -CF_{3}$   
 $R'_{F} = C_{2}F_{5}$  and  $R'_{F} = -CF_{5}$   
 $R'_{F} = C_{2}F_{5}$  and  $R'_{F} = -CF_{5}$ 

$$R'_F = C_3F_{7-}$$
 and  $R'_F = -CF_3$   
 $R'_F = C_3F_{7-}$  and  $R'_F = -C_2F_5$   
 $R'_F = C_3F_{7-}$  and  $R'_F = -C_3F_7$ 

This is simply not true based on the art disclosed as available by Caporiccio. Furthermore, in Example 1 (column 6, lines 6-17) of Caporiccio, "a perfluoropolyether liquid having the average formula (d) wherein the end groups  $R_F$  including mainly  $-CF_3$  and  $-C_2F_5...$ " is used. This disclosure again indicates that a pure, clean material that fits only one exact formula is not used; in other words Caporiccio is <u>not</u> able to independently select the  $R_F$  groups;

likewise, Moggi discloses the same six formulas (1-6; column 3, lines 6-47) as Caporiccio [(c-g); column 3/4, lines 58-68 and 1-12) for the perfluoropolyethers, but Moggi uses  $R_f$  and  $R'_f$  for the perfluoroalkyl end groups having 1 to 3 carbon atoms. However, Moggi gives no further indication except through art disclosed as available from Montedison (more recently Ausimont), DuPont, and Daikin as to exactly how many carbon atoms are in the end groups  $R_f$  and  $R'_f$  for each of the perfluoropolyethers. Thus, it is also my learned opinion that no indication is given here that one can independently select the end groups so as to give a pure material without other alternate end groups being present;

furthermore, from my 15+ years experience of collaborative research with DuPont, I have not known DuPont to produce any perfluoropolyethers comprising the repeat unit of structure 4 in Moggi et al. [same as structure (c) in Caporiccio] where both R<sub>f</sub> and R'<sub>f</sub> groups contain three carbon atoms essentially free of any one or two carbon groups, except for the said discovered perfluoropolyethers disclosed and claimed in the above-identified patent application by Jon L. Howell et al.; and

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful

false statements may jeopardize the validity of the application or any patent

issuing thereon.

Joseph S. Thrasher

Date